

# Steels for Vacuum Carburizing and Structure of the Carburizing Layer after Low Pressure Carburizing

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## I. General comments on application of low pressure carburizing technology for hardening constructional steel.

Low pressure carburizing, as well as atmospheric carburizing is a technology applied to parts made of low-carbon steel. Usually this is steel with C content within the range of 0,1-0,25%; sometimes, however, steel with higher content of carbon, even up to 0,4% is subject to carburizing.

After carburizing, parts undergo quenching and tempering, usually low-temperature tempering. It is more and more customary that quenching is followed by cold treatment and then tempering.

The purpose of such a combined treatment is to obtain variable hardness of the section of machine parts undergoing this process- the highest in the edge layer and the lowest in the core, as well as to get a favourable stress pattern, namely, the compressive stress in the edge layer and the tensile one in the core.

In practice, quality requirements for parts being quenched refer to surface hardness, agreed upon thickness of the quenched layer (for example, defined by the distance from the edge to the depth with the hardness of 550 HV), core hardness, microstructure of the layer and the core as well as deformations of parts.

Generally, the result of hardening of carburized parts is affected by:

- chemical composition of steel,
- carbon profile in the carburized layer,
- hardenability of steel and the carburized layer,
- temperature and time of austenitizing before quenching,
- intensity and a method of cooling down.

### Chemical composition of steel under analysis

Table 1 presents types and chemical composition of some steel samples used by Seco/Warwick for technological trials. The trials were carried out both for alloy steels as well as carbon steels. The alloy steel group covered steels with low hardenability, for example 5120, higher – 8620 and great hardenability, for example 8910.

Table 1 Composition of some steels used for carburizing or carbonitriding

Steel	Composition, % mass							
	C	Mn	Si	Pmax	Smax	Ni	Cr	Mo
<b>Carbon steels</b>								
1010	0.08-0.13	0.30-0.60		0.035	0.045			
1019	0.15-0.20	0.70-1.00		0.035	0.035			
1524	0.19-	1.35-		0.035	0.035			

	0.29	1.65						
<b>Alloy steels</b>								
<b>Cr-Mn</b>								
5120	0.17- 0.22	0.70- 0.90	0.20- 0.35	0.035	0.040		0.70- 0.90	
<b>Cr-Mn-Mo</b>								
4118H	0.17- 0.23	0.60- 1.00	0.20- 0.35	0.035	0.040		0.30- 0.70	0.08- 0.15
<b>Cr_Mn-Mo-Ni</b>								
8620	0.18- 0.23	0.70- 0.90	0.20- 0.35	0.035	0.040	0.40- 0.70	0.40- 0.60	0.15- 0.25
<b>Cr-Ni</b>								
3310	0.08- 0.13	0.45- 0.60	0.20- 0.35	0.025	0.025	3.25- 3.75	1.40- 1.75	
<b>Mo-Ni</b>								
4620	0.17- 0.22	0.45- 0.60	0.20- 0.35	0.035	0.040	1.65- 2.00		0.20- 0.30
4820	0.18- 0.23	0.45- 0.60	0.20- 0.35	0.035	0.040	3.25- 3.75		0.20- 0.30
<b>Cr-Mo-Ni</b>								
4320	0.17- 0.22	0.45- 0.65	0.20- 0.35	0.035	0.040	1.65- 2.00	0.40- 0.60	0.20- 0.30
8910	0.08- 0.13	0.45- 0.60	0.20- 0.35	0.025	0.025	3.00- 3.50	1.00- 1.40	0.08- 0.15

Generally, our experience leads to the following conclusion:

- application of low pressure carburizing for parts made of alloy steels gives very good results provided, however, that parts undergoing hardening have a section adjusted to hardenability of the steel and the carburized layer. High pressure gas cooling, due to smaller intensity as compared to oil cooling, can be used for parts with smaller sections or for parts with higher hardenability.
- surface quenching of parts made of low hardenability steel, for example carbon steels, performed by means of low pressure carburizing and using machinery for low pressure carburizing does not lead to satisfactory results. Positive results are in this case ensured by applying the technology of low pressure carbonitriding. Nitrogen in the presence of carbon has a synergizing effect on the increase of hardenability of diffusion layer, at the same time making its hardening easier.

### **Choosing a method of hardening and the temperature and time of soaking**

The technology of direct hardening starting with the carburizing temperature or undercooling from a lower temperature is applied most frequently.

Direct hardening is simple in terms of technology and guarantees that the optimum surface concentration of carbon obtained during carburizing process is maintained.

Undercooling causes that strains due to hardening decrease, mainly because of the smaller difference of temperatures in the beginning and at the end of the process. When carbon concentration in the carburized layer is higher than the eutectoid one, then during undercooling, due to lowering of carbon solubility in austenite, carbides may precipitate in the carburized layer, which leads to decrease in the amount of retained austenite after

hardening. Owing to a big size of austenite grains, carbides precipitate at grain boundaries, causing brittleness.

In order to reduce the size of austenite grain pearlitizing process can be applied after which the charge can be soaked up to the temperature of austenitizing and then hardened after soaking.

In order to ensure the highest possible hardness of the core of the parts, the temperature of soaking before hardening should be within the austenitic range. Hardening from lower temperatures will result in decrease of hardness, due to emergence of ferrite in the structure. Soaking time should ensure chemical homogeneity of austenite grains.

### **Effect of intensity and a cooling method**

A cooling rate of the charge should be higher than the critical speed in order to ensure formation of martensitic structure.

Higher cooling rates contribute to greater deformation of parts undergoing hardening.

High pressure gas cooling contributes to, in comparison to oil cooling, lowering the deformation of parts.

The cooling process ensuring a temperature stop around the  $M_s$  temperature, within the time necessary for the temperature to get stabilized on the section of the parts under hardening, has a significant effect on decrease and stabilization of deformations.

The technology of quenching of carburized steel characterized for their high hardenability, that means steel of significant alloyability, requires application of subquenching in order to lower/ or eliminate retained austenite and to obtain a satisfactory hardness profile.

### **What carbon profile after carburizing?**

Carbon profile should ensure the highest carbon concentration within the edge layer and then this concentration should be gradually dropping towards the core of parts. For traditional constructional parts such as shaft, gear wheel, carbon concentration at the edge should be eutectoid or slightly higher, yet not lower than 0,60%. Such carbon concentration ensures hardness after hardening within the range of 58-62 HRC.

Carbon concentration in the eutectoid point is lowered by alloy additives, in most cases it is lowered by Ti, and then by Mo, W, Si, Cr, Mn, Ni. For comparison, carbon eutectoid concentration in the 5120 steel amounts to 0,72%, and in case of the 8910 steel it is 0,41%.

Eutectoid carbon concentration guarantees, that after adequately quick cooling, a martensitic structure will form in the edge layer with contents of retained austenite lower than 10%.

Carbon concentration much above the eutectoid one increases contribution of retained austenite in the structure and lowers the related hardness. When this concentration goes above the boundary solubility in austenite, then carbides appear in the structure.

On the other hand, when carbon concentration in the edge layer is lower than 0,6% the martensite formed after hardening is too soft to ensure that the hardness of the edge layer is above 58 HRC. The additional factor contributing to lowering the hardness is the decrease of the edge layer hardenability which is connected with a lower carbon concentration.

In case of low pressure carburizing, during soaking in a vacuum chamber a sublimation of alloy components from the edge layer of parts takes place, this especially concerns manganese which also causes that the layer hardness lowers.

The carbon profile in the carburized layer of a defined alloyability as well as the intensity of cooling down decide about thickness of the hardened layer. In case of a less intensive cooling in order to obtain a comparable thickness of the hardened layer the carburized layer must be thicker.

In case of strict requirements concerning strength, the core structure of parts should also be martensitic. In other cases it can be martensitic- bainitic and even pearlitic-ferritic.

## **II. Low Vacuum Carburizing special and high-alloy steels**

Currently, it is common to search for and apply new materials, especially in automotive and aerospace, in order to limit the weight and improve the performance and durability of the end product. The features of these materials, characterized by an unconventional chemical composition, are ideally suited to precision heat treatment carried out in vacuum furnaces. Gear wheels, pinions, drive system bearings, pump bearings, camshafts and other parts in flight control equipment are most often made of high-alloy and special steel that underwent vacuum carburizing. Final qualities are ensured by hardening often connected with deep-freeze (cryogenic) treatment and tempering. Due to high, changing loads and/or operation in high temperatures, parts that are subject to treatment require layers that are 0.25-6.5 mm thick hardened case with a strength and ductile core. The time necessary for making such thick layers can be reduced by high temperature carburizing. Oil quenching and more frequent hardening in chambers provided with high pressure gas cooling ensures conversion of austenite into martensite in the carburized layer and the core. The optimum utility features of the parts under treatment depend on both the carbon profile in the layer as well as on the microstructure, which is a function of carbon profile, steel alloy additions, conditions of cooling while hardening and parameters of deep-freeze (cryogenic) treatment and tempering. The characteristic feature of the microstructure is the fact that in the hardened layer, apart from martensite, there appear carbides, the kind, number, size and morphology of which affect utility features of the parts. Steel vacuum carburizing is not an equilibrium process, thus in order to determine the process parameters expert systems are employed, such as FineCarb [1] technology, which, based on mathematical models and computer simulations as well as on the grounds of experimental verification can run the process with a very high accuracy and repeatability

This paper presents some of the results of vacuum carburizing by this method along with high pressure gas quenching of selected special and high-alloy steel obtained in actual trials.

### **Special and high-alloy steel grades**

The main alloy components of special steel is chromium Cr (11-18% of the mass) and molybdenum Mo (0,5-4.5%) and also, depending on the steel grade: nickel Ni (0,2 -4% of the mass, but also 6-13%), vanadium V (0.1-1.5%) and in some cobalt Co (10-18% of the mass). In case of high-alloy steel grades the main alloy component is molybdenum Mo (3-5% of the mass) or alternately wolfram W, chromium Cr (0,2-4.5%), nickel Ni (0,2-3.5%) vanadium V (0,1-1,5%).

Table 2 presents several examples of advanced types of steel. The majority of them have low carbon content and the required utility features are obtained through carburizing and hardening or heat treatment at all.

*Table 2. Examples of advanced special steel types used on high-load parts [2].*

Material	Chemical composition, mass %							
	C	Mn	Cr	Ni	Mo	Si	V	others
XD15NW	0.37	-	15.5	0.20	1.80	-	0.30	-
X13VDW	0.12	-	11.5	2.50	1.60	-	0.03	-
N360	0.33	0.50	15.0	0.40	1.00	-	-	-
IsoExtra								
R350	0.14	0.30	4.25	3.50	4.30	0.18	-	-
CBS-600	0.19	0.60	1.45	-	1.00	1.10	0.06	0.06 %AL
Pyrowear 53	0.10	0.35	1.00	2.00	3.25	1.00	0.10	2.0 %Cu
Pyrowear 675	0.07	0.65	13.0	2.60	1.80	0.40	0.60	-
CSB-50NIL	0.13	0.25	4.20	3.40	4.25	0.20	1.20	-
CSS-42L	0.12	-	14.0	2.00	4.75	-	0.60	12.5 %Co
Ferrium C61	0.15	-	3.50	9.50	1.10	-	0.09	18.0 %Co

**Vacuum  
carburizing  
LPC**

The vacuum furnaces (figure 1) were equipped with a low pressure, FineCarb, carburizing system. This system ensures thermochemical process within the temperature range of 800-1100°C (1470-2010°F) and within the pressure range of 0.5-10 mbar [3].

This process of low pressure carburizing is based on alternating and cyclic application of carburizing - boost and diffusion phase – soaking on the temperature [4]. During carburizing cycles the carburizing atmosphere in a given amount and time at the pressure range of 0.5 –10 mbar is provided to the process chamber. The carburizing atmosphere is a mixture of three gases (acetylene, ethylene and hydrogen), the composition of which was developed and patented by SECO/WARWICK and the Technical University of Łódź [5]. Chemical reactions taking place in the atmosphere and the catalytic effect of the charge surface lead to the formation of active carbon atoms, which are absorbed by austenite, saturating it and then diffusing inside the material. Dosing of the carburizing atmosphere is performed through precise mass flow controllers (MFC) ensuring repeatability of the settings and flows. The line of MFCs is provided with built-in appropriate pressure sensors and cut-off valves – manual and electromagnetic.



Figure 1. Vacuum furnace, LPC + HPGQ  
Type 15.0 VPT-4050/48N

The supply of the carburizing mixture in the diffusion cycles is held, whereas the pressure in the chamber is lowered by the pumping system to the level of 0.05 mbar or controlled at the level of partial pressure by means of nitrogen. At the same time, as a result of carbon

diffusion out of the edge zone inside the material, the carbon concentration on the charge surface is lowered and the thickness on the carburized layer increases. The example of the parameters and their course in the carburizing process is illustrated in figure 2.



Figure 2. FineCarb low pressure carburizing process cycle

The carbon profile in the layer being carburized on a particular steel type and in a chosen temperature range depends on a number and the length of carburizing and diffusion cycles. The process ensures that the carburized layer obtained is uniform even on the oddly shaped details; this process provides the capability of carburizing deep holes. The process is clean (no creation of hydrocarbons in a form of soot, resin or tar).

The carburizing system goes with a computer software, SimVac, which enables design of carburizing parameters of the typical steel used after carburizing, so that the details can get the required carburized layer, most frequently defined by its thickness and surface carbon concentration, figure 3. The control and supervision system is fully automated which leads to repeatability of the processes and to improvement of quality of heat treatment.

### Hardening in HPGQ vacuum furnaces

Special and high-alloy steels have a high hardenability due to a large content of alloy additives, so that they can be hardened efficiently in nitrogen under 15 bar pressure. Single-chamber furnaces already provide sufficient cooling speed (300-600 W/m<sup>2</sup>K) to obtain the right structure of the carburized layer and the core of the parts under treatment [6]. Gas quenching has many advantages in comparison with the traditional oil hardening:

- Environmentally friendly
- Labor safety,
- Decreased investment and operational expenses via shorter manufacturing cycle due to the elimination of the washing process after oil quenching
- Reduction of post heat treatment activities.

An important benefit is the potential to minimize deformation through an optimum selection of the cooling speed and direction as a result of controlling the pressure of the gas and its velocity and the area of inflow. Hardening with an isothermal stop, which is especially important in the case of treating large details can be achieved easily.

These features are available as standard in a single-chamber vacuum furnace, class LPC+HPGQ (Low Pressure Carburizing + High Pressure Gas Quench) that makes this equipment especially useful for heat treatment of special and high-alloy steel grades.

### **Technical tests**

A series of trials were performed in the SECO/WARWICK R& D facility [7] using Ferrium C61, CSB-50NiL and other advanced carburizing steel grades. Some of the results obtained are presented below. Thermochemical processes were carried out in a single chamber vacuum furnace – figure 1, which allows carburizing, hardening by means of a high pressure gas quench and tempering.

The results of the thermochemical treatment were evaluated by measuring profile of case hardness and carbon concentration, and changes of microstructure in the cross-section of the tested samples Ø 25x10 mm (1.0x0.4”) and 25x 150 mm (1.0x6.0”). Transverse microsections of the samples Ø 25x10mm (1.0x0.4”) were used for measuring changes of hardness HV0.1 or HV0.5 and microstructures, and rollers of Ø 25x150mm (1.0x6.0”) were used for evaluating carbon concentration at the depth of the carburized layer. Hardness profile was measured with a microhardness tester FM-700 (Future-Tech) and surface hardness was measured with Wilson Wolpert Testor 751. The microstructure was observed by means of an optical microscope Neophot and the scanning microscope Jeol 5600 LV, while metallographic specimen were digested with Nital, Adler’s reagent. Carbon concentrations at particular depths were measured with Leco tester by burning chips 0.05mm (0.002”) thick, which were taken by turning rollers Ø 25x150 mm (1.0x6.0”) previously tempered at the temperature of 650°C (1200°F).

### **Material Ferrium C61 [8]**

An advanced steel grade with improved utility features is the alloy Ferrium C61 (Questec Innovations).

This alloy belongs to a new group of martensite steel grades used for bearings and power transmissions. Ferrium C61 is an excellent alternative to typical materials used in gears, where the parts are required to have improved mechanical properties, but are not possible to be redesigned. Ferrium C61 was especially elaborated to ensure a high hardness of carburized surfaces (60-62 HRC) and possesses good tribological properties, ductility and fatigue resistance similar to the properties of typical steel grades used in such applications as AISI 9310 and EN36C, and apart from that it is also possessed with additional very high core strength.

Ferrium C61 was developed especially for high temperature carburizing. Heat treatment of this alloy is closely related to thermochemical processes, especially to vacuum carburizing. This provides the opportunity of hardening directly from the carburizing temperature using inert gas under high pressure. After cooling down to room temperature deep freeze treatment is advisable in order to make it possible for the martensite transformation to complete.

The alloy is usually tempered at a temperature of 482 °C (900°F); in this temperature the alloy shows a very good heat resistance. If it is required, nitriding can be applied, which makes it possible to obtain surface hardness of approximately 70HRC (1100 HV).

Carburizing Ferrium C61, then hardening and tempering causes precipitation of high dispersion carbide  $M_2C$  in the

carburized layer. This results in high hardness and surface compressive stress with a very low carbon content in metallic matrix. The low carbon content ensures at the same time, that secondary carbides decreasing ductility will not remain in the matrix. Applying the final shot blasting of details increases additionally fatigue resistance.

Ferrium C61 has a higher fatigue resistance than many other alloys available in the market. Strength tests proved, that it shows the fatigue resistance that is 15% higher than in case of the steel grade EN36C.

An example of heat treatment of Ferrium C61 is the presented data coming from the process no 0345 and 0348. Process no 0345 and 0348 was run according to the following parameters:

#### Process 0345

- Carburizing at the temperature of 1000°C (1832°F)
- Total time of carburizing segments: 10 minutes
- Total time of diffusion segments: 4 hours 15 minutes
- Direct hardening from the carburizing temperature: 1000°C (1832°F)
- Nitrogen hardening under pressure of: 5 bar
- Tempering: 17 h at 485°C (905°F)

#### Process 0348

- Carburizing at the temperature of 1000°C (1832°F)
- Total time of carburizing segments: 8 minutes
- Total time of diffusion segments: 4 hours 15 minutes
- Direct hardening from the carburizing temperature: 1000°C (1832°F)
- Nitrogen hardening under pressure of: 5 bar
- Tempering: 17 h at 485°C (905°F)

The results obtained are presented below in figure 4 and 5. Figure 4 illustrates hardness profile on the cross-section of hardened layer, whereas figure 5 shows microstructure. The case obtained was 1mm thick with the surface hardness within the range of 650-720 HV. It also illustrates the influence of the carbon concentration on the hardness obtained and microstructure. Too much concentration of carbon at the surface layer – process 345, in fact has an effect on the increase of hardness, but on the other hand, increases the amount of retained austenite and favors the occurrence of carbides  $M_7C_3$  on the austenite grain boundary.

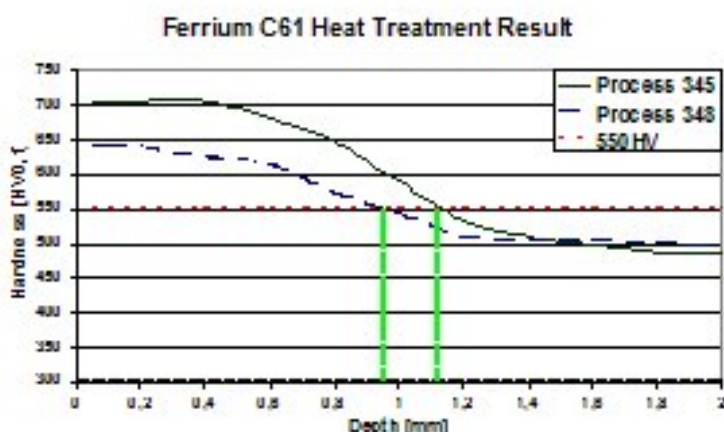
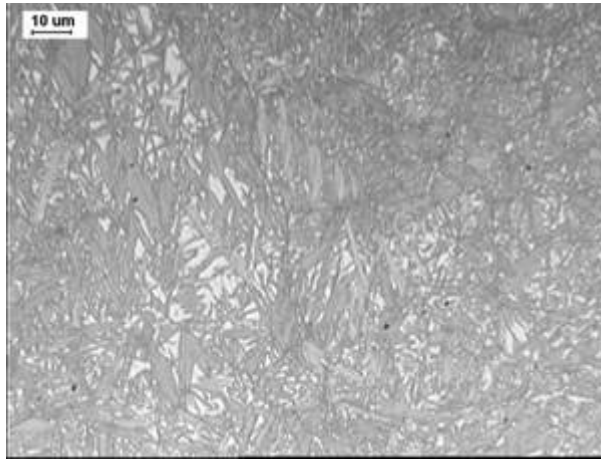
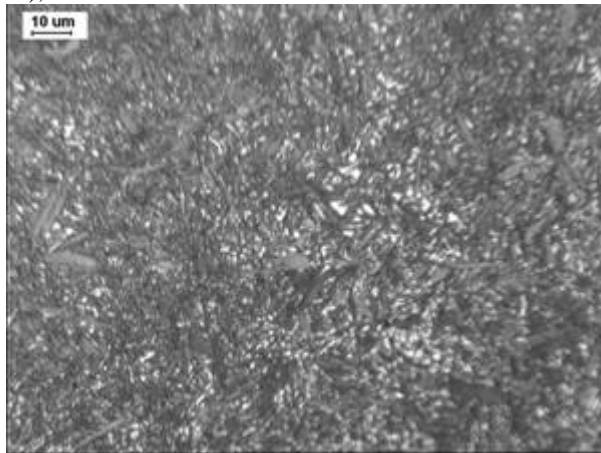


Figure 4 – Distribution of hardness HV 0.1 on the cross section of the hardened layer of Ferrium C61 specimens, thermochemical treated in the processes 345 and 348



*Figure 5a – Microstructure of the case of Ferrium C61 specimens after the process 0345*

Carbon martensite, precipitation hardened, carbides on austenite grain boundary, increased contents of retained austenite (light field), 0.73% C on the surface



*Figure 5b – Microstructure of the case of Ferrium C61 specimens after the process 0348*

Carbon martensite, precipitation hardened; minimum contents of retained austenite; 0.55% C on the surface.

### **Material CSB-50 NIL**

CSB-50 NIL steel (The Timken Company) shows properties very similar to those possessed by Ferrium C61. This is a special alloy designed for carburizing, used for bearings and gear transmissions, meant for operation at the temperatures ranges over 316°C (600°F). The chemical composition is selected so that the surface properly carburized and heat treated should in its microstructure have a small-sized tempered martensite with a significant contribution of carbides uniformly distributed. The main advantage of this steel grade in comparison with the commonly used hardened steel M50 is a ductile core.

Heat treatment after carburizing can be done following the method as in case of high-speed steel, that means hardening and several processes of high tempering, it is also acceptable to apply a deep freeze (cryogenic) treatment.

*Table 2. Nominal chemical composition of the steel grade CSB-50NIL, 6-5-2 and the grade M50, % of the mass*

Steel grade	Chemical composition, mass %								
	C	Mn	Cr	Ni	Mo	Si	V	Co	W
CSB-50NIL	0.13	0.25	4.20	3.40	4.25	0.20	1.20	-	-
6-5-2	0.24	0.30	4.20	-	5.0	0.30	1.90	-	6.0
M50	0.80	0.25	4.13	0.10	4.25	0.40	1.00	0.12	-

Steel CSB-50 NIL has successfully been used in the most reliable bearings in aircraft engines. This steel grade or similar can be also used for cutting tools with excellent cutting ability and ductility of the core. An example of such a tool can be screw-taps. Table 2 below lists a chemical composition of CSB-50 NIL and the steel grade 6-5-2 (steel grades of the alloyability of the high-speed steel M50).

An example of heat treatment of CSB-50NIL designed for bearing elements is the process 0464 data. The process was run according to the following parameters:

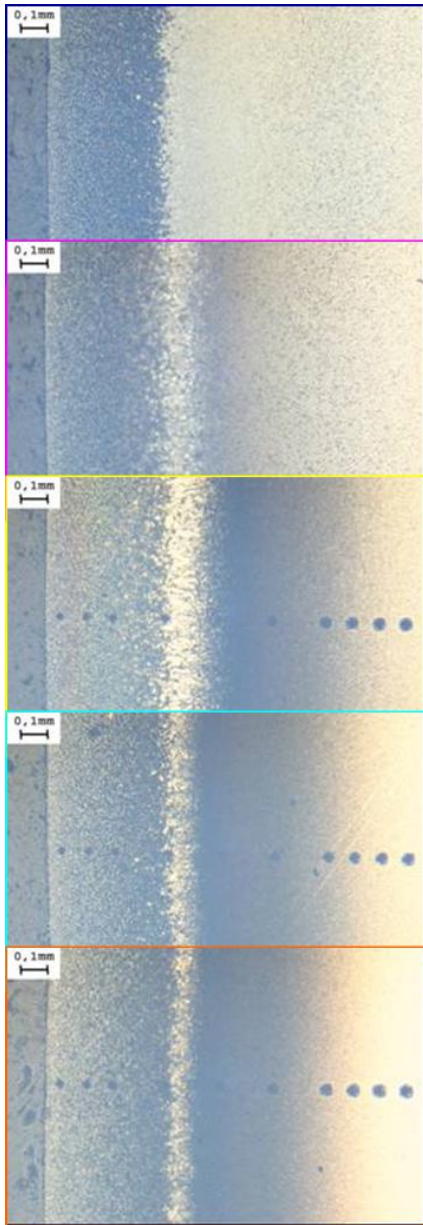
- Carburizing at the temperature of 950°C (1742°F)
- Total time of carburizing segments: 1 hour 19 minutes
- Total time of diffusion segments: 5 hours 50 minutes
- Direct hardening from the carburizing temperature: 950°C (1742°F)
- Nitrogen hardening under pressure of: 9,5 bar
- I tempering 2 hours at 200°C (392°F)
- II tempering + 2 hours at 520°C (968°F)
- III tempering + 2 hours at 520°C (968°F)
- IV tempering + 2 hours at 520°C (968°F)

Table 3 as well as figures 6 and 7 present the results obtained. Case hardness distributions HV0.5 – figure 7 and microstructure changes after different stages of treatment – figure 6, and the surface hardness HRC – table 3.

Process stage	Carburizing and hardening	+ I tempering 200 C (392°F)	+ II tempering 520 C (968°F)	+ III tempering 520 C (968°F)	+ IV tempering 520 C (968°F)
HRC	57	55	62	61	60

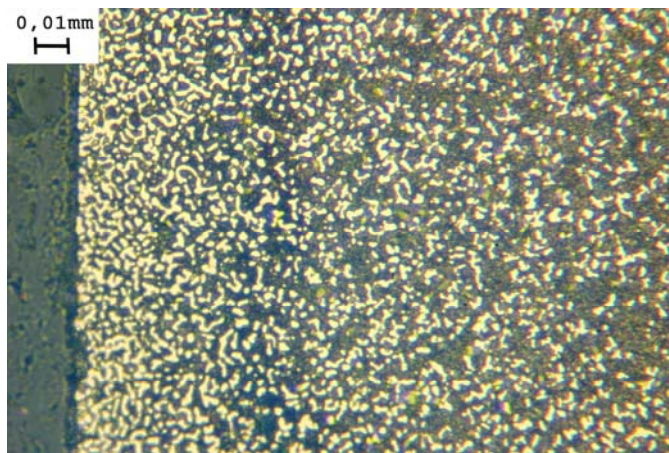
As a result of carburizing, a great amount of carbides were obtained in the carburized layer along with high contribution of retained austenite, especially at the depth of 0.4 -0.5 mm (0.016-0.020”) – figure 6. It is reflected by a hardness change on the section of the carburized and hardened layer, where at the depth of 0.5 mm (0.02”) a significant decrease of the hardness value can easily be seen – figure 7.

Applying high tempering at the temperature of 520 °C (968°F) several times led to almost a complete removal of retained austenite as well as to an increase of the layer hardness up to the level of 750 HV and its thickness getting stabilized at 0.5 mm (0.02”).



*Figure 6a – Comparison of the CSB-50 NIL steel structure obtained after successive stages of the heat treatment under 0464 process*

*Characteristic white belt is a result of the increased amount of retained austenite. The amount of retained austenite lowers along with the amount and intensity of tempering process. (after hardening on the top and below after I, II, III, IV tempering)*



*Figure 6b – Comparison of the CSB-50 NIL steel structure obtained after successive stages of the heat treatment under 0464 proces. Carbides against a background of the tempered martensite*

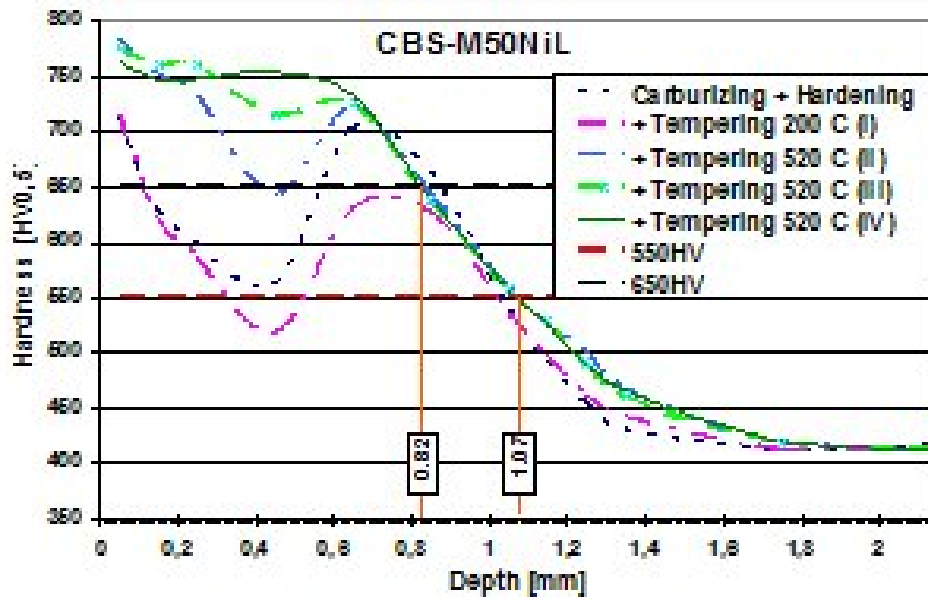


Figure 7- Distribution of the case micro-hardness HV 0,5 of the CSB50 NIL steel after the 0464 process

#### Material 6-5-2

Figures 8, 9 illustrate results of the vacuum carburizing technology of low-carbon steel with the alloyability of the high-speed steel 6-5-2 (table 2) used for cutting tools such as screw-taps. The purpose of the treatment was to harden screw-taps in such a way so that the external layer could be very hard, yet maintaining good ductility. It was assumed that such properties would be ensured by a carburized layer with the carbon surface concentration similar to the concentration in a typical high-speed steel M50, guaranteeing its secondary hardening after quenching and high tempering [9,10] and the agreed upon thickness  $h_{550}$  equal to 1,1-1,2 mm (0.043-0.047”).

Cylindrical screw-taps  $\Phi$  8 mm (0.315”) underwent thermochemical treatment consisting of vacuum carburizing, gas quenching and tempering. The heat treatment was carried out in two stages. The first stage was carburizing and the second stage was quenching and tempering under the conditions as those applied for the steel grade 6-5-2.

- Carburizing parameters:
- Carburizing at the temperature of 1040°C (1904°F)
- Total time of carburizing segments: 27 minutes
- Total time of diffusion segments: 2 hours 31 minutes
- Cooling down with the furnace to 550°C (1022°F) and continuing in nitrogen to the ambient temperature.

Parameters of hardening and tempering:

- Gradual heat-up with stops at: -900°C (1652°F) – 10 minutes  
-1100°C (2012°F) – 15 minutes  
-1175°C (2147°F) – 35minutes
- Nitrogen quenching under the pressure of 4,5 bar
- Tempering: 2x 120 minutes at 560°C (1040°F)

Figure 8 shows the results of the case hardness distribution, whereas figure 9 the results of observation of the microstructure. As it is shown in figure 8 the hardened layer can be characterized by its high surface hardness – approximately 900-950 HV0.1 and effective case depth of  $h_{700}=1.0$  and  $h_{550}= 1.1$  mm, whereas the material core of screw-taps shows hardness of approximately 300-350 HV0.1.

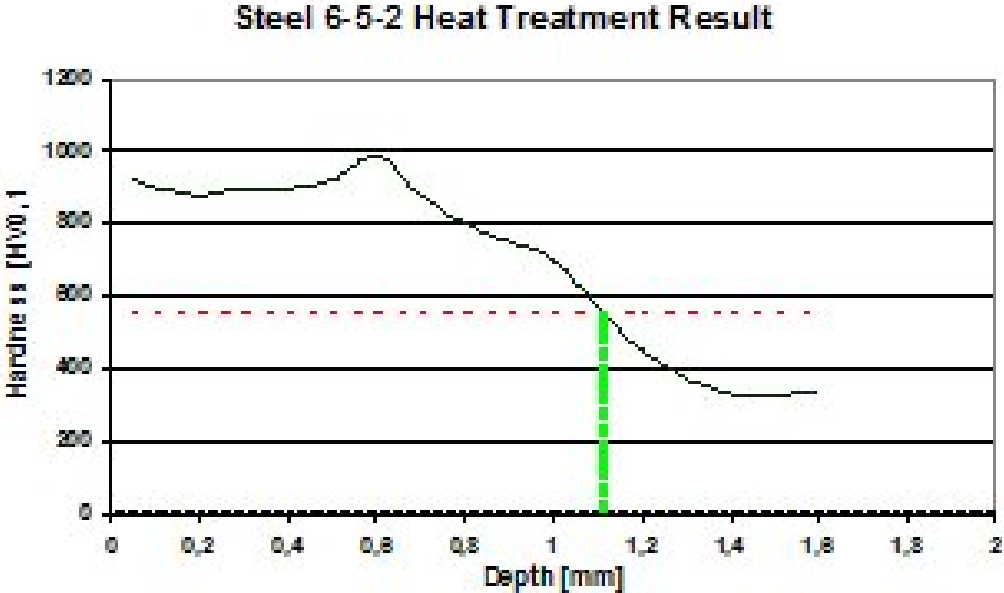
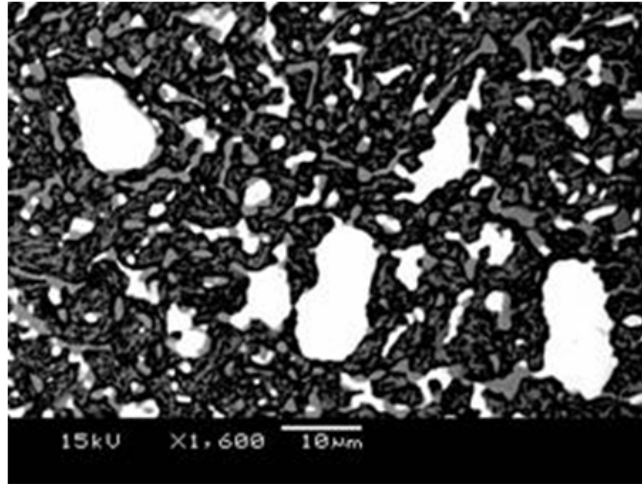


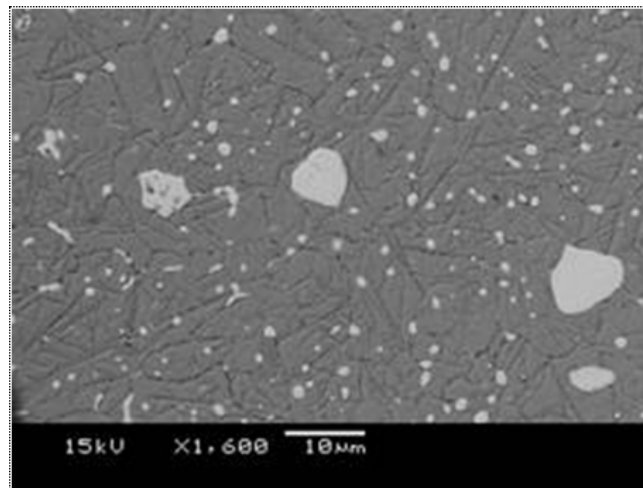
Figure 8 – Case hardness profile on the cross section of the screw-tap,  $\Phi 8$  mm (0.315”), 6-5-2 steel.

Vacuum carburizing of low-carbon high-speed steel 6-5-2- has a great effect on the increase of carbon concentration in the case, which is performed by the increase of the carbide layer [9, 10]. At the stage of carburizing, the output carbides grow in the over-saturated austenite and new carbides form, namely the carbides of MC type based on VC carbide and  $M_6C$  carbides with characteristic branched adhesions – figure 9a.

Vacuum heating, especially heating during austenitizing for hardening causes dissolution of the released carbides and carbon diffusion into the layer – figure 9b. As a result of hardening in the carburized layer there forms a structure composed of different contributions of martensite, retained austenite and undissolved carbides. High tempering at the temperature of  $560^{\circ}C$  ( $1040^{\circ}F$ ) of the case causes its secondary hardening and transformation of the retained austenite into martensite, which in cooperation with the existence of coagulated, uniformly distributed carbides gives a high hardness – figure 8.



*Figure 9a.. Microstructure of the case of the screw-tap,  $\Phi 8$  mm (0.315"), 6-5-2 steel. Case, after carburizing at  $1040^{\circ}\text{C}$  ( $1904^{\circ}\text{F}$ ) – white and gray carbides shown at the background of the austenite matrix*



*Figure 9b. Case, after hardening from  $1175^{\circ}\text{C}$  ( $2147^{\circ}\text{F}$ ) and double tempering at  $560^{\circ}\text{C}$  ( $1040^{\circ}\text{F}$ ) – white inclusions of carbides at the background of the tempered martensite with retained austenite.*

#### **Material X5CrNiMo17-12-2 [9,10]**

Chromic-nickel corrosion resistant steel grades are used for parts that are required to be corrosion resistant and possess a high mechanical strength. An example of such a steel grade application in new areas of machine industry can be products such as ball screws and nuts. So far such elements were manufactured following the technology of low-alloy steel carburizing and the required corrosion resistance was ensured by cadmium plating. Cadmium plated layers are nowadays regarded as harmful. Carburizing of steel grades that are corrosion resistant is an original solution combining high mechanical strength and corrosion resistance of the core with the output product that is a thin, hard carburized layer with a desired corrosion resistance [11].

An example of this application of vacuum carburizing of the special steel X5CrNiMo17-12-2 with a nominal chemical composition shown in table 4 is presented below.

*Table 4. Nominal chemical composition of X5CrNiMo17-12-2 (Bohler), % of the weight*

Material	C [%]	Mn [%]	Cr [%]	Ni [%]	Mo [%]	Si [%]
X5CrNiMo17-12-2	0,03	1,00	17,5	11,5	2,25	0,50

For the purpose of these tests, cup shaped samples of  $\Phi 20 \times 10$  mm (0.79x0.39) with the wall 2 mm (0.08") thick were used. The samples were surface quenched by means of a single-segment vacuum carburizing at the temperature of 1050°C (1922°F) – carbon saturation time of 2 minutes. Cooling was performed in a vacuum chamber by taking the samples out of the heating chamber.

The layer obtained shows the surface hardness at the level of 900 HV 0,1 and case thickness of  $h_{550}=40-50\mu\text{m}$ - figure 10. The layer microstructure contains a great number of small- sized carbides released in the martensite-bainite-pearlite-austenite matrix – figure 11.

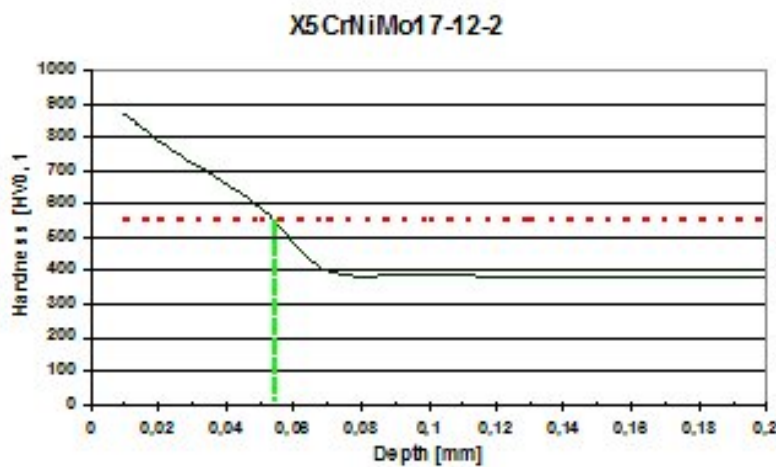


Figure 10- Hardness distribution on the cross section of the carburized layer of the steel X5CrNiMo17-12-2

In the process a thin, hard layer composed of  $M_7C_3$  and  $M_{23}C_6$  was formed.

Selection of the process parameters: temperature, time, cooling speed can affect on a wide range the structure formed with its features, especially the number of the carbides released as well as the composition of the matrix.

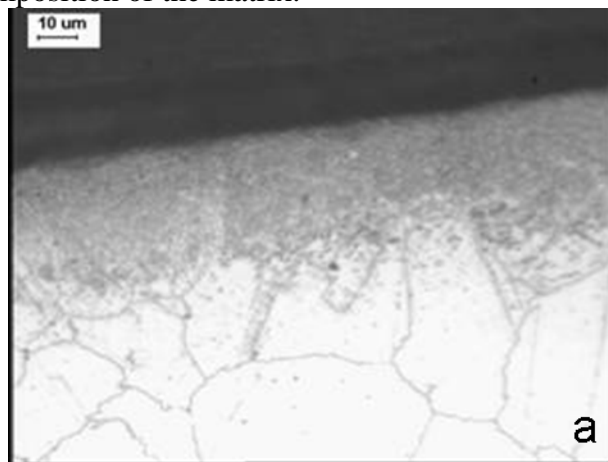


Figure 11a –Photographs of the microstructure of the carburized layer of the steel X5CrNiMo17-12-2. Case after 2 min carburizing at 1050°C (1922°F)

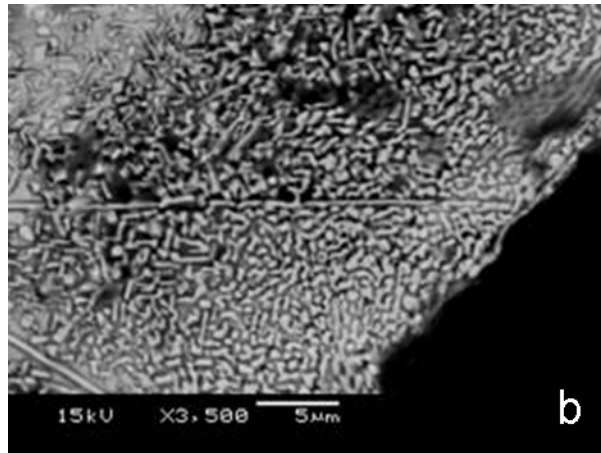


Figure 11b. The carburized layer –very small releases of carbides can be observed on the edges as well as inside grains of metallic matrix. The matrix is composed of: in the dark area – martensite, bainite, pearlite and retained austenite; in the light area – austenite.

### Conclusion

The above results were obtained during test trials of vacuum carburizing (LPC) special and high-alloy steel grades in vacuum furnaces equipped with FineCarb® process technology demonstrate the capability of obtaining parts case and core of a given structure and features.

Use of modern steel grades makes it possible to obtain improved mechanical properties of the surface layer, such as, for example: increasing surface hardness related to carbide forming, or increasing the acceptable temperature of operation of carburized elements as well as obtaining anticorrosive layers.

The additional advantage of high-alloy steel grades is their better hardenability, which allows hardening at lower cooling speeds, which in turn, causes less distortion of parts. Ideal for such types of applications are single-chamber vacuum furnaces with high pressure gas quenching (HPGQ).

The advantage of applying this technology of thermochemical treatment is the capacity to run the process at high temperatures, which reduces treatment time significantly, yet increases the production efficiency.

### References

1. P. Kula, J. Olejnik, J. Kowalewski: "FineCarb® – The Smart System for Vacuum Carburizing". *Heat Treating & Hardening of Gears conference 16.03.2004, Chicago*.
2. Frederick J. Otto, D H. Herring: "Vacuum carburizing of aerospace and automotive materials". *Heat Treating Progress, January/February 2005s. 33 – 37*.
3. P.Kula, J. Olejnik: „Some Technological Aspects of Vacuum Carburizing”. *Proc. of the 12<sup>th</sup> International Federation of Heat Treatment and Surface Engineering Congress. Melbourne. 2000. Vol. 3.195-220*.
4. Patent application PL347192, P. Kula, J. Olejnik, P. Heilman: „Mehtod for carburizing of steel parts under oxygen free, under-pressure atmosphere”.
5. Patent application US20060102254, P. Kula, J. Olejnik, P. Heilman: „Hydrocarbon gas mixture for the under-pressure carburizing of steel”. 2006.07.02.
6. R. Gorockiewicz, M. Korecki, L. Maldziński, J. Olejnik: „State and perspectives of gas quenching on the base of Seco/Warwick experiences in heat treatment of tool and HSLA steels, and FineCarb® vacuum carburizing”. *Nowoczesne technologie w inżynierii powierzchni, Łódź-Spała październik 2006*.
7. A.Adamek, M. Korecki: „Seco/Warwick Internal Data Base - R&D Department”, 2003-2005;

8. *Questek Innovations: „Ferrium C61 – Case Hardened Gear Steel with Ultrahigh Strength Core”, October 2003.*
9. *R. Gorockiewicz, J. Olejnik: „Case hardened structure of high speed steel”. Archiwum Odlewnictwa, 2004, R. 4, nr 11, T. 1, s. 155.*
10. *R. Gorockiewicz: „Morphology of carbides and case carburized layer of high speed steels”. Inżynieria Materiałowa, nr 5 (147), rok XXVI, wrzesień-październik 2005, s. 483.*
11. *T.Turpin, J. Dulcy, M.Gantois: Carbon Diffusion and Phase Transformations during Gas Carburizing of High-Alloyed Stainless Steels: Experimental Study and Theoretical Modeling. Metallurgical and Materials Transactions A, Vol. 36A, october 2005, pp 2751-2760*